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Methods of Testing Solid Dielectrics

PART I. Preparation of Samples.

By the Engineering Department, Aerovox Corporation

IN SEVERAL types of scientific work, it becomes necessary to measure the electrical characteristics of solid dielectric materials. Such tests include dielectric constant, power factor, dielectric strength, Q, and insulation resistance.

Examples of solid dielectrics checked constantly in industrial testing laboratories are glass, paper, ceramics, textiles, plastics, mica, and rubber. There are many others. Some of these materials, and the composite products into which they enter, are hard and unyielding; others are pliable.

The technique of preparing a sample piece of dielectric material for electrical tests follows an established pattern. The material is arranged, in one of several ways, as the dielectric portion of a test capacitor. Electrical measurements then made upon the capacitor indicate certain characteristics of the dielectric itself.

The peculiar problems attendant to preparation of solid dielectrics commend this subject to special attention.

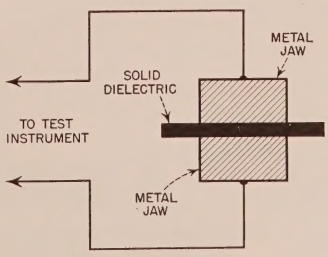
BASIC METHOD

The simplest method of arranging a flat plate or sheet of dielectric material for electrical tests suggests the arrangement shown in Figure 1. Here, the dielectric is held tightly between parallel-ground metal jaws (with or without a guard electrode) which form the capacitor electrodes. The jaws are connected to an appropriate test instrument, such as a bridge, capacitance test oscillator, or megger. A sample of dielectric tubing might be arranged in a similar fashion with a tight-fitting inner metal plug and snug outer metal sleeves as electrodes.

The inadequacy of this arrangement is due to the fact that the electrode faces do not rest upon *all* portions of hard dielectric surfaces (See exaggerated view in Figure 2), and, as a result, air voids occur in the surface "valleys" along the dielectric faces. In some solid dielectrics (such as soft materials) the valleys may be eliminated through pressure when the jaws are tightened. When voids are squeezed out in this fashion, however, the thickness of the material is altered and the jaws must be provided with a micrometer screw to show true thickness.

MERCURY ELECTRODES

To offset the shortcomings of the solid-jaw test jig just described, mercury electrodes have been suggested for testing electrical characteristics of hard, solid dielectric materials. One method, which has been employed to some extent in testing laboratories, is shown in Figure 3.



SIMPLE METAL-JAW TEST CAPACITOR

FIG. 1

In this arrangement, the dielectric body is floated on the surface of mercury held in a suitable dish. The second capacitor plate of mercury is floated on top of the dielectric sample and is confined by a thin, short cylinder of non-conducting material. End-immersed wire connections are run from the two bodies of mercury to the test instruments.

Our own experience with this system, both with the arrangement shown in Figure 3 and with special mercury-contact jigs, indicates that it is not adaptable to large-lot sampling where speed is a factor. Moreover, the mercury soon picks up foreign particles, such as dirt, dielectric flakes, and the like, and must be cleaned or replaced. Air tends to be trapped in small pockets on the dielectric surface by the weight of the mercury, and the latter does not wet the dielectric surface, therefore possibly failing to get down into the minute depressions along the surface. At the same time, the continuous handling of mercury may introduce a health hazard.

A better scheme seems to be attachment of a separate, intimately-bound metal plate to each face of the dielectric sample. The test capacitors thus obtained may then be clipped into a special low-loss test jig, such as shown in Figure 4, the jig being

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plugged directly into the test instrument input circuit.

There are in current use several methods of preparing dielectric samples in this manner. The principal ones are (1) silvering by painting with silver pastes and firing at high temperatures; (2) silvering by painting with special silver pastes which "metallize" on air drying; (3) silvering by means of chemical deposition; (4) attaching metal foils by means of adhesives; (5) metallizing by evaporation in vacuo; and (6) metallizing by cathode sputtering. These systems will be discussed separately.

HIGH-TEMPERATURE SILVERING

In this process each face of the dielectric body is painted in the shape of the desired electrode plate with a special silver paste. The latter material, in a common form, is a mixture of silver oxide and silver tartrate (in both true solution and colloidal dispersion) held together by a suitable gum or other binder.

The painted body is then roasted at a temperature in the vicinity of 1000° F. During this step of the process, both the silver oxide and the silver tartrate are reduced to metallic silver. Liberation of oxygen from the oxide accelerates reduction of the tartrate and aids in burning away the binder. The firing process requires up to 15 minutes for completion as commonly carried out.

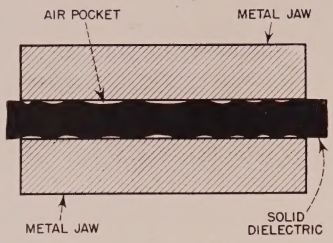
Obviously, the formation of silver electrodes in this manner is restricted to dielectric materials capable of withstanding the intense heat of the roasting process. Not only must the material not burn up, but its electrical characteristics must not be altered by the firing.

ROOM-TEMPERATURE SILVERING

There have appeared recently several electrical silver paints which do not require firing at elevated temperatures. These compositions appear to be suspensions of finely-divided metallic silver in an organic binder. They are applied to the dielectric surface by brushing or spraying and dry by solvent evaporation after various lengths of exposure to open air at room temperature.

We have tested certain of these materials but have not, up to this writing, found them as satisfactory for dielectric testing as the silver paints that must be fired. Following is a brief resumé of our observations:

Air-drying silver composition brushed on a large number of first-grade mica films and dried at room temperature showed lower Q and higher power factor values (both indications of poor quality) than films of the same quality silvered by painting and firing. The power factor of mica is improved by drying in the



EXAGGERATED CROSS-SECTION OF DIELECTRIC FILM BETWEEN JAWS

FIG. 2

firing ovens. On a basis of average values obtained by testing a large number of samples, power factor of mica silvered with the air-dried composition appeared to be only half as good as mica of the same grade silvered by firing. On the same basis, the Q value would appear to be only one-sixteenth as good. The dielectric constant, however, exhibited not more than 0.2% difference. Guided by power factor and Q figures, this mica might have been rejected needlessly as inferior had the test with air-dried silver electrodes been unchecked.

Some slight improvement was obtained by copper plating the silver surfaces electrically in an acid $CuSO_4$ bath and subsequently washing and drying the samples. The improvement, however, was too slight to justify the additional operations.

SILVERING BY CHEMICAL DEPOSITION

Solid dielectric materials may be silvered chemically by the process employed in making mirrors. Basically, this consists of suspending the dielectric in a solution of a silver salt (such as ammoniated silver nitrate) and depositing metallic silver by addition of a reducing agent, such as Rochelle salt or formaldehyde.

This process has been much improved. Present use of triethanolamine for the slow reduction of acidified silver nitrate, for example, has reduced the detonation hazards of the older processes.

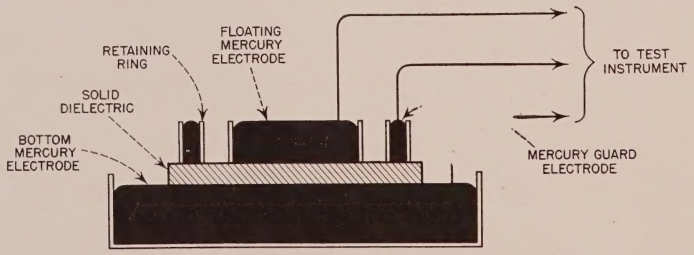
Chemical deposition of silver has several disadvantages. While it is rapid and requires no heat, its use is limited to non-porous dielectrics, because of the chemical bath. Paper, textiles, and certain plastics cannot be treated. Chemically-deposited silver coats are also relatively thin (triple coatings made in our laboratory averaged 0.000025 inch). Success of the process demands extraordinary cleanliness of the dielectric surface, a condition which is most difficult to obtain. Portions of the surface not to be metallized must be masked carefully with wax or other material incapable of reacting with the plating chemicals. All samples must be washed and dried thoroughly before electrical tests can be made.

These disadvantages, accompanied by certain physiological inconveniences occasioned by the process, plus the inability to obtain the close metallizing control afforded by other processes of silvering, have lessened the extent to which this method is employed. Our own laboratory studies have shown little difference in observed electrical characteristics of first-grade mica, non-porous ceramics, and glass silvered by chemical deposition against the same dielectrics silvered by painting and firing.

ATTACHMENT OF FOIL PLATES

It is convenient to attach thin tinfoil or aluminum-foil plates to the parallel surfaces of dielectric samples. Thinly-spread Petrolatum is usually employed as an adhesive.

In order that a true picture may be obtained of the electrical characteristics of the dielectric material, only the absolute minimum of Petrolatum is permissible between foil and dielectric surface. Moreover, the dielectric surface and the adhesive must be free of all dust and other foreign particles, and every wrinkle must be smoothed carefully out of the foil, in order to remove air and grease pockets. Actual handling of the materials must be reduced to a



CROSS-SECTION OF MERCURY-TYPE TEST CAPACITOR WITH DIELECTRIC SAMPLE

FIG. 3

minimum because of the detrimental effect of hand perspiration and oils.

More than ordinary care is necessary in the preparation of dielectric samples in this manner. But even this is not a guarantee that Petrolatum-attached foils will enable a true indication of electrical characteristics of the dielectric. The very thinnest film of the adhesive still will influence results somewhat.

In a recent laboratory study, we compared results obtained by attaching foils and by silvering (painting and firing). Dielectric constant and Q, both at 1 Mc., were the characteristics studied. A large number of films of first-grade mica were employed as the dielectric.

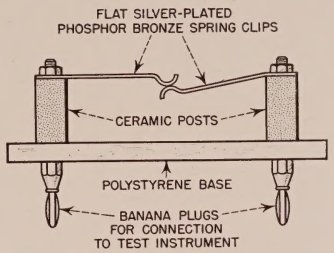
The coded mica films were cleaned by degreasing, foils were attached with great care by means of Petrolatum, and K and Q were tested. The foils then were removed, the films degreased to remove every trace of Petrolatum, and silver was applied by painting and firing. (Area of silvered electrode was identical with that of foil). The samples then were checked again for K and Q.

Dielectric constant of the silvered films was from 1.03 to 1.75 times that of the same films with attached foils. Q of the silvered films varied from 1.04 to 15 times that of the same films with attached foils.

ELECTRICAL METHODS OF DEPOSITING METAL PLATES

Two well-known methods of metallizing solid dielectric materials are *evaporation* and *sputtering*. These are illustrated in Figure 5.

Figure 5-A shows the arrangement of apparatus for evaporation. Several pieces of dielectric material to be plated are arranged a short distance from a metallic filament within an evacuated chamber. The filament is



SPRING-CLIP "LOW-LOSS" TEST JIG

FIG.4

coated with the metal which is to be plated.

In operation, the chamber is evacuated and the filament is raised slowly to incandescence by passing a current through it, and held at that point for a few minutes. The surface metal of the filament is evaporated by the process and is deposited upon the dielectric bodies.

Careful masking is necessary to prevent undesired metallizing of certain portions of the dielectric surface.

Figure 5-B shows the arrangement of apparatus for cathode sputtering. Here again, a highly-evacuated chamber is employed. (Pressure is reduced to 0.001 mm.). The cathode, usually in the form of a flat parallel plate, is made of the metal to be plated upon the dielectric surface and is connected externally to one terminal of a 10,000-volt power supply. If d.c. is employed, the cathode is connected to the negative terminal. The anode, usually made of aluminum, is connected to the positive terminal.

The body to be plated is mounted between anode and cathode and not more than 25 mm. from the latter. When the high voltage is applied, the discharge phenomenon carries metal from cathode to the dielectric surface where it is deposited.

Both evaporation and sputtering have the advantage that they are rapid, do not require elevated temperatures or chemical solutions, and may be carried on in restricted quarters. Like the chemical deposition of silver, however, it has the disadvantage that masking of the dielectric is required. Only one surface may be plated at a time, as the process is normally carried out; the dielectric surface must be very clean; and a very high vacuum (obtained only with the aid of a diffusion pump) is necessary.

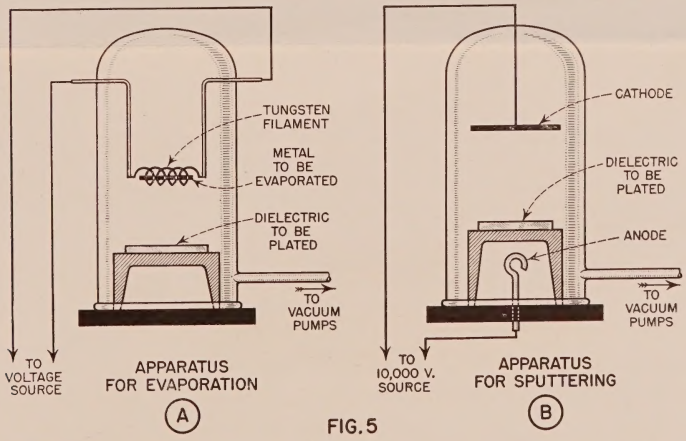


FIG.5

COMPARISON OF METHODS

Our studies of dielectric preparation would indicate that silvering by high-temperature firing is the most effective method for heat-resistant materials when high-vacuum equipment is not available. This method is applicable to large-lot sampling. For all-around use with all solid dielectrics, porous and non-porous, plating by evaporation or cathode sputtering is perhaps the most desirable when large-lot sampling is not a factor and when high-vacuum equipment is easily obtainable.

When neither of these methods may be employed, non-porous solid dielectrics may be plated by chemical deposition, although this method is time-consuming and will not permit a large number of samples to be tested quickly.

Where single samples, or a very few samples are to be tested and the length of testing time is unimportant, mercury electrodes may be employed.

Foil plates may be attached to dielectric samples when sufficient time is available for the extraordinary care required in the use of adhesives and when conditions do not permit use of any of the other methods. At best, this method may be fully depended upon only for comparative measurements.

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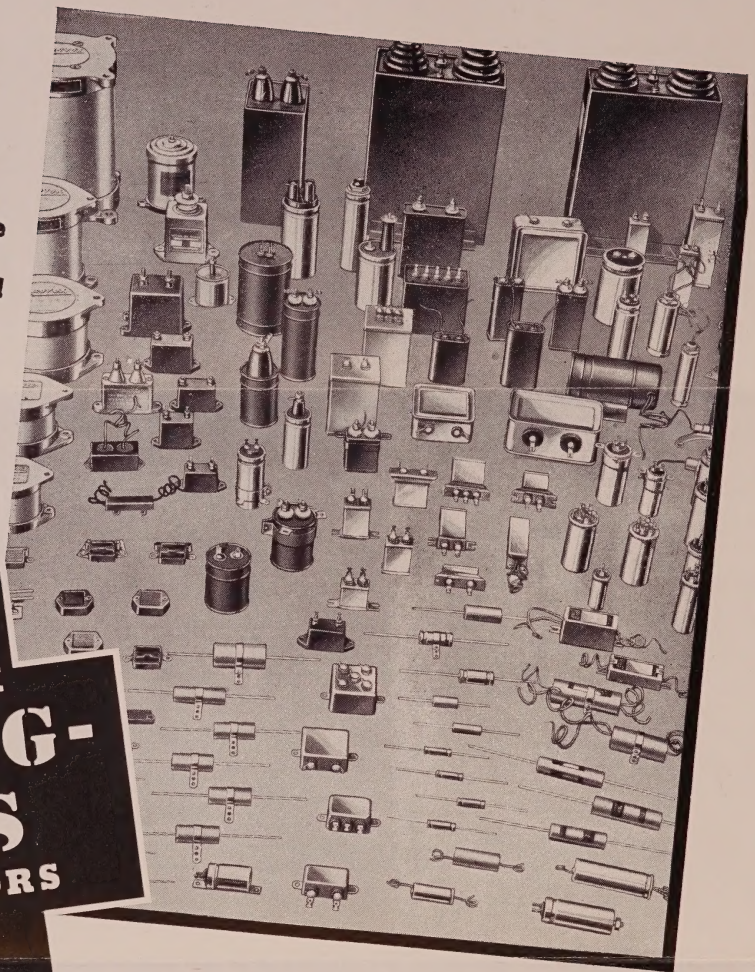

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